

The Effect of Simplified Bonding Agents on the Bond Strength to Dentin of Self-Activated Dual-Cure Resin Cements

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By
Anita M. Shade, BDS, DDS


Dunn Dental Clinic
JBSA-Lackland, TX

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**The Effect of Simplified Bonding Agents on the Bond Strength to Dentin of
Self-Activated Dual-Cure Resin Cements**

Anita M Shade

APPROVED:



Col Craig S. Vandewalle



Col Michael Wadjowicz



Maj Clifton Bailey

26 Apr 2013

Date:

APPROVED:



Col Thomas R. Schneid

Dean, Air Force Postgraduate Dental School

**Uniformed Services University
of the Health Sciences**

Manuscript/Presentation Approval or Clearance

INITIATOR

1. USU Principal Author/Presenter: Capt Anita Shade
2. Academic Title: Resident, Advanced Education in General Dentistry Residency (AEGD-2)
3. School/Department/Center: Air Force Postgraduate Dental School (AFPDS)
4. Phone: 210-671-9822
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CHAIR OR DEPARTMENT HEAD APPROVAL

1. Name: Kraig S. Vandewalle, Director, Dental Research
2. School/Dept.: AFPDS / AEGD-2
3. Date: 6 June 2013

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Chair/Department Head Approval: Kraig S. Vandewalle Date 6 June 2013

SERVICE DEAN APPROVAL

1. Name: Col Thomas Schneid



2. School (if applicable): AFPDS

3. Date: 7 June 2013

4. __Higher approval clearance required (for University-, DoD- or US Gov't-level policy, communications systems or weapons issues review").

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VICE PRESIDENT FOR EXTERNAL AFFAIRS ACTION

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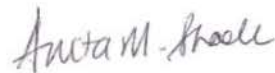
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A handwritten signature in cursive script, reading "Anita M. Shade".

ANITA M SHADE
Advanced Education in General
Dentistry Residency
Uniformed Services University
Date: **6 June 2013**

DEDICATION

*"For whoever finds me finds life
And obtains favor from the Lord".*

Proverbs 8:35

I dedicate this to my Lord and Savior Jesus Christ, who is my source of hope and inspiration- who equips me daily with strength and blesses the work of my hands.

ACKNOWLEDGEMENTS

My sincerest thanks go to Dr. KS Vandewalle whose dedication to the cause of Dental Research has made all this possible. His ideas were the seeds that helped grow this project. Under his patient guidance, I was able to see the research process progress from start to finish. Far beyond materials and methods, hours of debating over data and mulling over results, his tutelage has helped spark an insatiable quest for scientific investigation in the hearts of novice researchers like myself.

I would like to acknowledge my training officer, Dr. M Wajdowicz, who trusted me and gave me all the leeway I needed as I juggled responsibilities at work and home. Sir, I want you to know your support is truly appreciated.

ABSTRACT

Objective: Due to known incompatibilities between self-cured resin cements and simplified dental adhesives, clinicians are compelled to maintain traditional multi-step adhesives in their inventory. The Kerr Corporation introduced NX3, dual-cure resin cement, that employs a unique redox initiator system that is reportedly compatible with simplified adhesives and obtains high bond strengths whether the dual-cure cement is light- or self-cured. To date, there is no literature to support this claim. It is the aim of this study to verify the accuracy of the claim so that clinicians can take advantage of its properties with the comfort of knowing it is supported by evidence-based dentistry. This study evaluated the shear-bond strengths to dentin of two dual-cure resin cements either in self- or dual-cure modes when used in combination with simplified or non-simplified adhesive agents.

Methods: One-hundred sixty human third molars were mounted in dental stone, sectioned with a low-speed diamond saw (Isomet) to expose the dentin surface and finished with 600-grit sandpaper. The mounted specimens were divided into four groups of forty teeth based on dentin treatment with four dental adhesives: two simplified adhesives, Prime and Bond NT (Dentsply) and Adper Prompt L-Pop (3M/ESPE); and two non-simplified adhesives, Optibond FL (Kerr) and Clearfil SE (Kuraray). The four groups were further divided into four subgroups of ten, bonded with two different cements, NX3 (Kerr) or Calibra (Dentsply), using self- or dual-cure

activation. Adhesives were applied to dentin as per manufacturers' directions. The specimens were placed in the Ultradent Jig, the cement was mixed and inserted into the mold to a height of 3-4 mm and light cured for 20 seconds or allowed to chemically polymerize.

Specimens were stored for 24 hours in 37°C distilled water and tested in shear (Instron). A mean and standard deviation was determined per group (Appendix A). Data was analyzed with 2-way ANOVA/Tukey's post hoc test per cement type ($\alpha=0.025$) and bonding agent ($\alpha=0.0125$). Following testing, each specimen was examined using a 10X stereomicroscope to determine failure mode as adhesive, cohesive or mixed.

Results:

With both NX3 and Calibra, bond strengths significantly increased when specimens were dual cured ($p<0.025$). Also with either cement in either mode, the non-simplified dental adhesives performed the best ($p<0.025$).

Within the simplified dental adhesives, no significant difference was found between specimens bonded with either NX3 or Calibra in both cure modes ($p < 0.0125$).

The non-simplified adhesive, Clearfil SE, had more mixed fractures than the other adhesives. Dual-curing was associated with more mixed fractures.

Conclusions:

- When used specifically with simplified dental adhesives in either cure mode, NX3 does not produce significantly higher bond strengths than Calibra.
- In general, lower bond strengths continue to be observed when simplified dental adhesives are used with any resin cement in self-cure mode.
- Improved dentin bond strengths can be expected when the cement is dual-cured.

Figure 1 – Shear Bond Strengths (MPa) in Self-Cure Mode

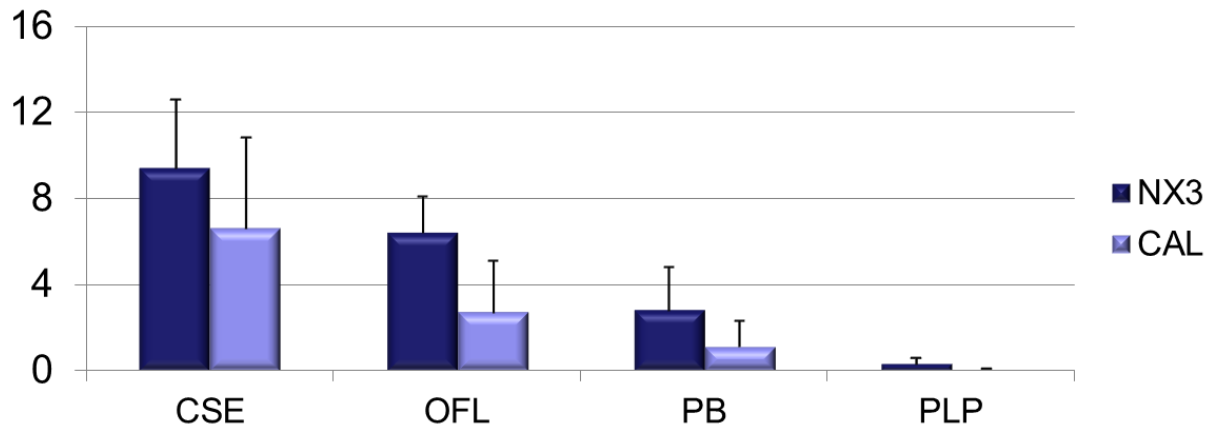


Figure 2- Shear Bond Strengths (MPa) in Dual-cure Mode

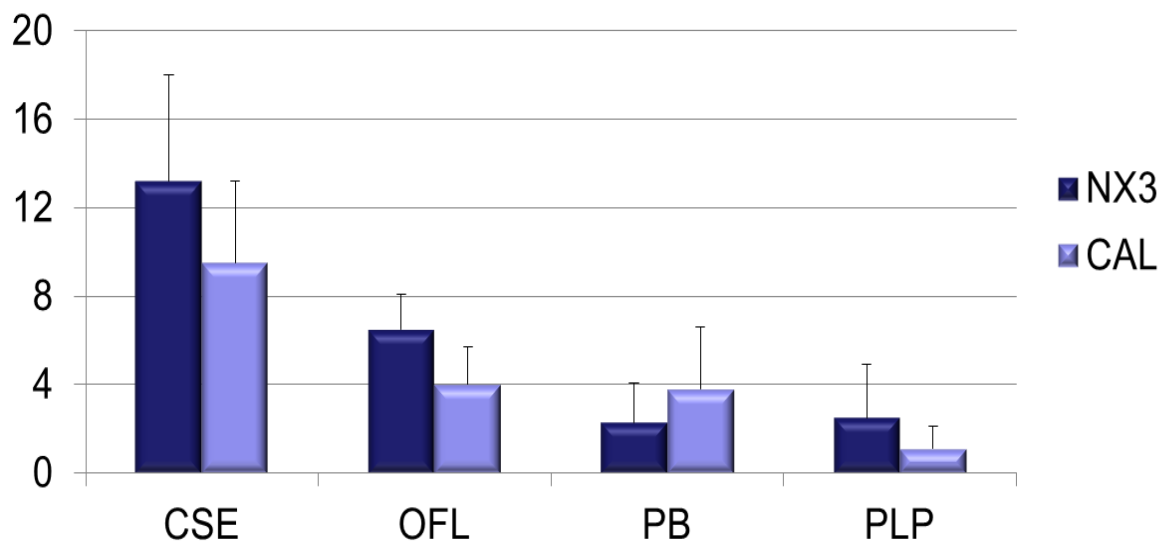


TABLE OF CONTENTS

| | Page |
|--|------|
| Title | i |
| Approval | ii |
| Dedication..... | iii |
| Acknowledgements | iv |
| Abstract | v |
| Table of Contents | ix |
| List of Figures | xii |
| List of Tables | xiii |
| I. BACKGROUND AND LITERATURE REVIEW | |
| A. The Birth of the Simplified Dental Adhesive | 1 |
| B. Effects of Simplification at the Microscopic Level..... | 2 |
| C. Incompatibility between Simplified Dental Adhesives and Self-Activated Dual-Cure Resin Cements | 4 |
| D. Development of NX3 | 6 |
| II. OBJECTIVES | |
| A. Objective Overview | 7 |
| B. Specific Hypotheses | 8 |

III. MATERIALS AND METHODS

| | |
|---|----|
| A. Experimental Design Overview | 9 |
| B. Experimental Design | 10 |
| C. Statistical Management of Data | 21 |

IV. RESULTS

| | |
|---|----|
| A. 2-Way ANOVA results Based on Adhesive..... | 22 |
| B. 2-Way ANOVA results Based on Resin Cements | 25 |
| C. Fracture Mode Results | 28 |

V. DISCUSSION

| | |
|---|----|
| A. Overview | 30 |
| B. Overcoming the Incompatibilities between Simplified Dental Adhesives and Self-Cure Resin Cements..... | 30 |
| I. Eliminate the Acid-Base Reaction..... | 31 |
| II. Reduce the effects of the Acid-Base Reaction: adequate light curing..... | 35 |

| | | |
|-------|---|----|
| III. | Eliminate the Oxygen Inhibited Layer..... | 35 |
| VI. | CONCLUSION..... | 39 |
| VII. | DISCLOSURES..... | 39 |
| VIII. | APPENDIX | |
| | A. Raw Data by Group | 40 |
| | B. Statistical Analysis | 49 |
| | Literature Cited | 57 |

LIST OF FIGURES

| | Page |
|---|------|
| Figure 1 Shear Bond Strengths (MPa) in Self-Cure Mode..... | viii |
| Figure 2 Shear Bond Strengths (MPa) in Dual-Cure Mode..... | viii |
| Figure 3 Adhesive Classification..... | 2 |
| Figure 4 The Mounting Process | 15 |
| Figure 5 Specimen Preparation | 16 |
| Figure 6 Shear Bond Strength Testing..... | 20 |
| Figure 7 Fracture Mode Results..... | 29 |
| Figure 8 Treatment Strategies Flow Chart | 38 |

LIST OF TABLES

| | Page |
|---|------|
| Table 1 Study Groupings..... | 13 |
| Table 2 Application Methods..... | 14 |
| Table 3 2-Way ANOVA Based on Adhesive | 24 |
| Table 4 2-Way ANOVA Based on Cement..... | 27 |

I. BACKGROUND AND LITERATURE REVIEW

A. The Birth of the Simplified Dental Adhesive

Ever since Buonocore described acid etching as a means to increase resin-enamel bond strengths over fifty years ago, we have been in constant pursuit of the ideal bonding agent (Buonocore, 1955). The initial etch-and-rinse adhesives required three steps that included an acidic conditioner, primer, and adhesive monomer. Examples include Optibond FL (Kerr) and Adper Scotchbond MultiPurpose (3M ESPE).

Over the years, the trend has been to develop systems that are “simplified”, or in other words, involve fewer steps with less procedure time (Tay et al, 2002). A simplified bonding agent is one in which the adhesive step is incorporated into the primer. Manufacturers began to combine the primer and resin monomer components to create a two-step or simplified etch-and-rinse agent. Examples include Optibond Solo Plus (Kerr), One-Step (Bisco), and Prime and Bond NT (Dentsply).

Self-etch adhesives have been an even more recent introduction where the use of an acidified primer has eliminated the use of the conditioner. An example of a popular non-simplified self-etch adhesive is Clearfil SE (Kuraray). Today, simplified versions of the self-etch adhesives on the market are one-step systems where the acidified primer and adhesive monomer are mixed together and placed in a single

step (Figure 3). Examples include Optibond All-in-One (Kerr), All-Bond SE (Bisco), Xeno 4 (Dentsply) and Adper Prompt L Pop (3M ESPE).

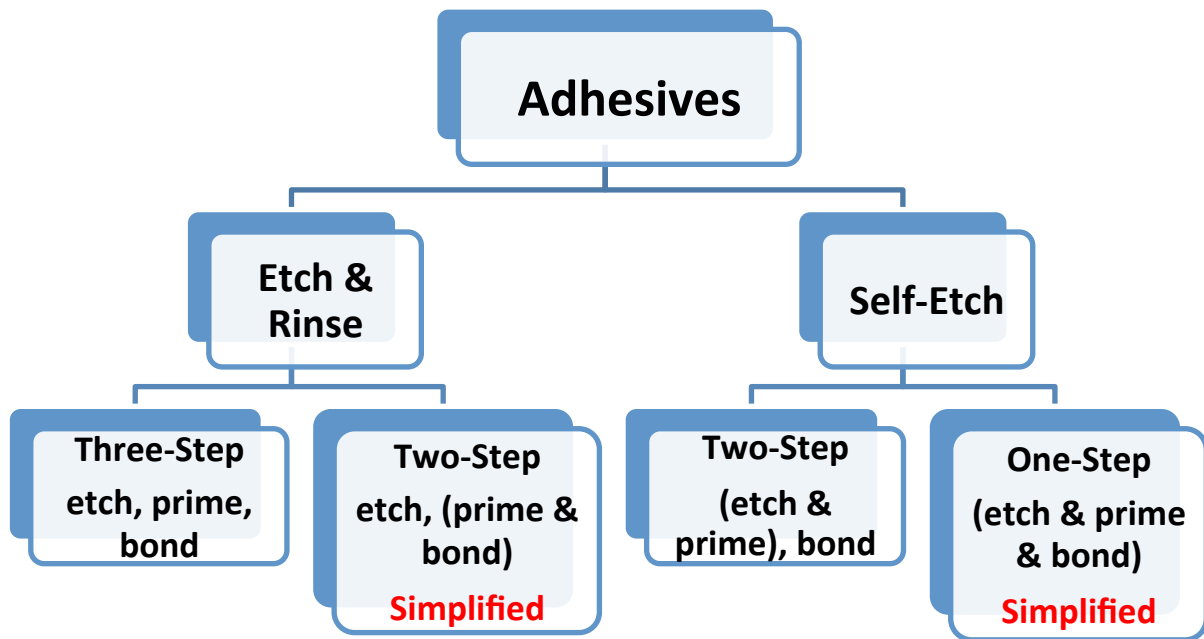


Figure 3 - Adhesive Classification

B. Effects of Simplification at the Microscopic level

Using restorative systems with simplified adhesives does not necessarily result in reduced bond strength to dentin (Moll et al, 2002; Armstrong et al, 2003). However, clinicians began to report bonding failures when self-cured “build-up” composites were bonded with simplified adhesive systems (Swift, 1999). They were alerted to potential incompatibilities between self-cured resins and certain bonding systems

(Hagge et al, 2001). Simplified systems used with light-cure resins were found to produce bond strengths that were considerably higher than when used with self-cure resins (Tay et al, 2003). The process of simplification involves incorporation of acidified resin monomers to the primer-adhesive combination. This results in a more hydrophilic mix. The concentration of acidic resin monomers is even higher in simplified self-etch adhesives where they serve to etch through the smear layer and enable bonding to the underlying dentin (Tay et al, 2004, 2003). The hydrophilic property improves the wetting of the demineralized collagen matrix (Eick et al, 1997). However, this layer acts like a semi-permeable membrane enabling the transudation of water from the underlying dentin across an osmotic gradient toward the oxygen inhibited bonding agent-resin cement interface (Tay et al, 2004). These are described as water trees and interfacial blisters under transmission electron microscopy and contribute to diminished bond strengths of self-cure composites when compared to their non-simplified counterparts (Tay et al, 2003).

More significantly, simplified adhesives can lead to the deactivation of the amine initiators in self- and dual-cure composites. Conventional self-cure composites utilize a binary redox initiator system that consists of benzoyl peroxide (BPO) with aromatic tertiary amines (Ryter IE, 1985). The amines react with the acidified monomers present in the superficial oxygen-inhibited layer and are unavailable to initiate the self-cure. This ultimately results in incomplete polymerization and compromised bond strengths along the composite-bonding agent interface (Bowen et al 1982; Ikemura et al, 1999; Nakamura et al, 1985).

C. Incompatibility Between Simplified Dentin Adhesive Agents and Self-Activated Dual-Cure Resin Cements

As mentioned earlier, the oxygen-inhibited layer in simplified adhesives acts as a hypertonic medium that triggers osmotic fluid transport through the permeable adhesive layer. It is also a source of acidic resin monomers that deactivate tertiary amines (Suh et al, 2003). The combination results in an incompatibility between self- and dual-cure composite resin materials when used with simplified adhesives as evidenced by lower bond strengths and presence of water blisters at the interface (Suh et al, 2003). The adverse chemical interaction between catalytic components of self-cured composites and simplified adhesives is the major cause of bond strength reduction whereas permeability of the adhesives to water causes only a minor reduction in bond strength (Tay et al, 2003). When the permeability component was completely removed, as with the use of neat water-free resins, even low concentrations of acidic monomers were shown to deactivate tertiary amines in self-cured resins (Suh et al, 2003).

Overall, the consequences are more acute in simplified self-etch than with simplified etch-and-rinse adhesives (Tay et al, 2003). Furthermore within the simplified etch-and-rinse adhesives, incompatibility was accentuated in some adhesives more than others (Swift et al, 2001). The decrease in tensile bond strengths of self-cure resins to dentin was shown to be inversely proportional to the acidity of the etch-and-rinse system (Sanares et al, 2001). In both of the above studies, the most acidic simplified

etch-and-rinse agent, Prime and Bond NT by Dentsply (pH- 2.68), had the weakest bond strengths when compared to the least acidic, One-Step by Bisco (pH- 4.60).

It should be noted that when a dual-cure cement is sufficiently light cured, the incompatibility does not occur (Tay et al, 2003). The bond strength to dentin is directly related to amount of light energy to which it is exposed (Takahashi et al, 2010).

Manufacturers of dual- or self-cure cement systems accept this incompatibility and indicate their use exclusively with non-simplified etch-and-rinse or self-etch adhesives.

“When used in self-cure or dual cure (limited or no light curing) techniques, Calibra Esthetic Resin Cement is contraindicated for use with Xeno-III Single Step Self-Etching Dental Adhesive. Chemical/product incompatibility may adversely affect product efficacy, leading to premature restoration failure”.

(Contraindications/precautions- Calibra Directions for use)

If a dual-cure cement is to be used with a simplified adhesive, adequate light curing of the cement is emphasized. Some manufacturers recommend use of an additional dual-cure activator. However, it has been shown that the use of activators does not completely eliminate this incompatibility (Tay et al, 2003).

D. Development of NX3

In 2007, the Kerr Corporation released a new dual-cure resin cement called NX3. They claim that the NX3 or Nexus third generation, employs a unique redox initiator system that is acid resistant and can initiate polymerization in the dark and in the presence of an acidic environment (Bui et al (Kerr), 2007/ 2008). Consequently they claim NX3 can be used with any adhesive system on the market without compromising bond strength. According to a press release from Kerr in Feb 2007: *“NX3 is compatible with self-etch and total-etch adhesives and obtains high bond strengths whether the cement is light or dual cured. A dual-cure activator for the adhesive is no longer needed and while light curing is always recommended even when there is limited light accessibility (i.e., PFM crowns); good adhesion is achieved in self-cure situations.”*

To date, there are no published articles in the literature to evaluate the credibility of the company's claims. According to researchers at the Kerr Corporation, when coupled with five commercial simplified dental adhesives in self-cure mode, NX3 showed the highest dentin shear bond strength values compared to three other dual-cure resin cements (Bui et al (Kerr), 2007/2008).

A few recent unpublished abstracts funded by manufacturing companies such as Dentsply (Liu et al, IADR 2010) ,Tokuyama (Hirata et al, IADR 2012) and Bisco (Chen et al, IADR 2013) that indirectly looked at NX3 in their study models, have not

shown improved dentin bond strengths with NX3 and simplified adhesive agents in self-cure modes.

II. OBJECTIVES

A. Objective Overview

The purpose of this study was to evaluate whether or not the proprietary initiators in NX3 are able to circumvent the incompatibility issue with simplified dental adhesives in self-cure mode. If they are, do they exhibit dentin bond strengths similar to those achieved in the dual-cure mode?

It was the aim of this study to verify the accuracy of the manufacturer's claim so that clinicians can take advantage of its properties with the comfort of knowing it is supported by evidence-based dentistry. This study evaluated the shear-bond strengths to dentin of two dual-cure resin cements NX3 and Calibra either in self- or dual-cure modes when used in combination with simplified or non-simplified adhesive agents.

B. Specific Hypotheses

This study tested two specific null hypotheses as follows:

1. There is no difference in the shear bond strength of NX3 or Calibra to dentin based on the choice of adhesive bonding agents, either simplified (Prompt L-Pop, Prime and Bond NT) or non-simplified (Optibond FL, Clearfil SE).
2. There is no difference in the shear bond strength to dentin of dual-cure cements, NX3 or Calibra, based on the curing mode, either dual- or self-cure

This study tested two alternative hypotheses as follows:

1. NX3 has higher shear bond strengths to dentin than Calibra when used with simplified adhesives like Prompt L-Pop and Prime and Bond NT.
2. Dual-cure cements have stronger shear bond strengths to dentin when activated in light-cure mode.

III. MATERIALS AND METHODS

A. Experimental Design Overview (Tables 2,3)

The resin cements chosen for this study are NX3 (Kerr) and Calibra (Dentsply). Calibra is a resin cement which can be used in light-, self- or dual-cure modes. However, the manufacturer contraindicates the use of Calibra in pure self-cure applications (limited or no light curing) with simplified self-etch or etch-and-rinse adhesives in the absence of a dual-cure activator. The manufacturers of NX3 claim complete compatibility with all adhesives irrespective of cure mode.

Four dental adhesives were utilized, two non-simplified- Optibond FL (Kerr) and Clearfil SE (Kuraray) and two simplified- Prime and Bond NT (Dentsply) and Prompt L Pop (3M ESPE). See Figure 3.

1. Resin Cement/Bonding Agent combinations:

- Prime and Bond NT-NX3/Calibra
- Prompt L-Pop-NX3/Calibra
- Optibond FL- NX3/Calibra
- Clearfil SE-NX3/Calibra

2. Activation modes: each of the above groups were further subdivided based on cure mode

- Dual-cure
- Self-cure

A total of 16 subgroups were created (see Table 1). Ten specimens were prepared per subgroup resulting in 160 total specimens. Group 1: Prime and Bond NT (subgroups #1-4); Group 2: Adper Prompt L- Pop (subgroups #5-8); Group 3: Optibond FL (subgroups #9-12); Group 4: Clearfil SE Bond (subgroups #13-16). Shear bond strength was tested after 24 hours of storage in distilled water at 37 degrees centigrade.

B. Experimental Design (Figures 4 - 6)

All samples were created by one provider to minimize inter-operator differences and to ensure uniformity of fabrication.

One hundred sixty extracted human third molars stored in 0.5% Chloramine-T at 4° C were used within 6 months following extraction. The teeth were mounted in dental stone in PVC pipes with the crown exposed and accessible (Figure 4). A diamond saw (Isomet, Buehler, Lake Forest, IL) was used to remove 2mm or more of coronal tooth structure to ensure dentin exposure and the proper orientation of the surface relative to the direction of the applied shear force. Each specimen was then examined under a stereomicroscope (SMZ-1B, Nikon, Melville, NY) at 10X magnification to ensure complete exposure of the dentin surface with no residual

enamel. A uniform smear layer was created on the flat dentin surfaces using ten passes on 600-grit carbide paper (Figure 5).

The 160 prepared teeth were randomly distributed to create four groups (40 specimens to each group) based on the four bonding agents used. The bonding agents were applied to the dentinal surface according to manufacturer's instructions (see Table 3). The adhesive was cured as recommended by the manufacturer using the Bluephase 16i (Ivoclar, Amherst, NY) light-curing unit. Irradiance of the curing light was monitored periodically with a radiometer (Bluephase Radiometer, Ivoclar) to verify irradiance levels remained above 1200 mW/cm².

Each of the four groups were further divided into four equal subgroups of ten specimens each with each subgroup tested with either one of the two resin cements being evaluated in either self- or dual-cure activation mode.

A priori power analysis helped confirm that a sample size was adequate. The sample size of 10 per group provided 80% power to detect a small effect size (0.26 or approximately 0.52 standard deviation difference among means for the main effect of bonding agent, and 0.23 or approximately 0.46 standard deviation difference between means for the main effects of cement type and cure mode) and similarly sized differences for the interaction terms when testing with a 3-factor ANOVA at the alpha level of 0.05 (NCSS PASS 2002).

The prepared specimens were placed in an Ultradent Jig and secured beneath the white non-stick Delrin insert (Ultradent, South Jordan, UT). The resin cement was then mixed and applied into the mold according to manufacturer's instructions (see Table 2) to a height of 3-4mm. The bonding area was limited to a 2.4mm diameter circle determined by the Delrin insert. The self-cure subgroups were allowed to self-cure undisturbed for a period of 15 minutes in a light-proof container (Figure 4). The dual-cure subgroups were bulk-light cured for 20 seconds to simulate light penetration achieved in a clinical setting. Following the application of the resin cement with the designated curing method, all specimens were stored for 24 hours in distilled water at 37 degrees centigrade. After 24 hours, the shear bond strength of all specimens was tested using the Instron 5543 testing machine (Instron, Norwood, MA) at a crosshead speed of 1mm/min using the notched blade at a ninety-degree angle (Figure 6).

Shear bond strength values in megapascals (MPa) were calculated from the peak load of failure (newtons) divided by the specimen surface area. The mean and standard deviation were determined for each group. The resultant data of the various groups was then analyzed to verify the three null hypotheses.

Following testing, each specimen was examined using 10X stereomicroscope to determine failure mode as either: 1) adhesive fracture at the cement/adhesive/dentin interface, 2) cohesive fracture in cement or dentin, and 3) mixed (combined adhesive and cohesive).

Table 1- Study Groupings

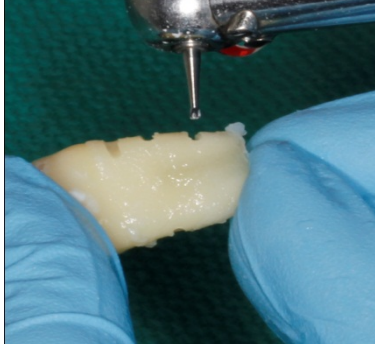
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|---------|---------------------|-------------|---------------------------|
| Group 1 | Prime and Bond NT | Subgroup 1 | NX3 + self-cure mode |
| | | Subgroup 2 | NX3 + dual- cure mode |
| | | Subgroup 3 | Calibra + self-cure mode |
| | | Subgroup 4 | Calibra + dual- cure mode |
| Group 2 | Adper Prompt L- Pop | Subgroup 5 | NX3 + self-cure mode |
| | | Subgroup 6 | NX3 + dual-cure mode |
| | | Subgroup7 | Calibra + self-cure mode |
| | | Subgroup8 | Calibra + dual-cure mode |
| Group 3 | Optibond FL | Subgroup 9 | NX3 + self-cure mode |
| | | Subgroup10 | NX3 + dual-cure mode |
| | | Subgroup11 | Calibra + self-cure mode |
| | | Subgroup12 | Calibra + dual-cure mode |
| Group 4 | Clearfil SE | Subgroup13 | NX3 + self-cure mode |
| | | Subgroup14 | NX3 + dual-cure mode |
| | | Subgroup 15 | Calibra + self-cure mode |
| | | Subgroup 16 | Calibra + dual-cure mode |

Table 2 - Application Methods

| Adhesive | Type | Manufacturer's application instructions |
|------------------------------|--|---|
| Prime and Bond NT (Dentsply) | Two-step, etch-and-rinse (simplified) | Etchant: apply and leave undisturbed (15s) Water rinse. Gently air dry (5s) Bond: Apply and leave undisturbed (20s) Gently air dry Light cure (10s) |
| Adper Prompt L-Pop (3M ESPE) | One-step, self-etch (simplified) | Mix the liquids in the red and yellow blister, brush the mixture onto tooth surface (15s) Gently air dry (5s) Light cure (10s) |
| Optibond FL (Kerr) | Three-step etch-and-rinse (non-simplified) | Etchant: apply and leave undisturbed (15s) Water rinse Gently air dry (5s) Primer: apply with light scrubbing motion (15s) Gently air dry (5s) Bond: Apply to a thin layer Light cure (30s) |
| Clearfil SE (Kuraray) | Two-step self-etch (non-simplified) | Primer: apply and leave undisturbed (20s) Gently air dry (5s) Bond: apply to a thin layer Light cure (10s) |
| NX3 (Kerr) | Resin Cement: dual-cure mode | Dual cure paste 2mm increments Light cure (20s) |
| | Resin Cement: self-cure mode | Dual cure paste Bulk fill |
| Calibra (Dentsply) | Resin cement: dual-cure mode | Mix equal lengths of base and catalyst for 20s 2mm increments Light cure (20s) |
| | Resin cement : self-cure mode | Mix equal lengths of base and catalyst for 20s Bulk fill |

Figure 4 – The Mounting Process

A- The extracted teeth notched prior to mounting



B- Stone poured into PVC pipes



C- Extracted teeth mounted in stone

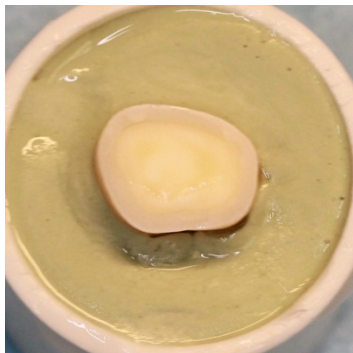


D- Storage in distilled water



Figure 5 – Specimen Preparation

A- Sectioned tooth specimen



B- Application of bonding agent as per manufacturers' instructions



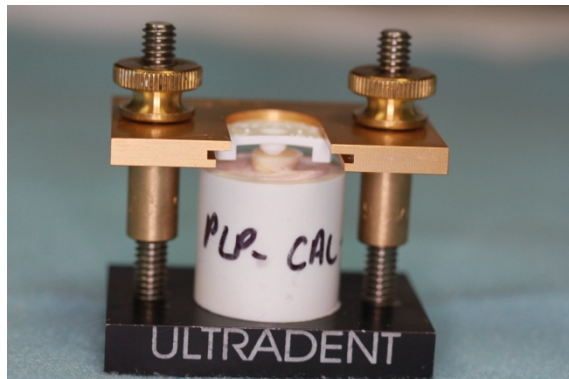
C- BluePhase 16i (Ivoclar) light curing unit



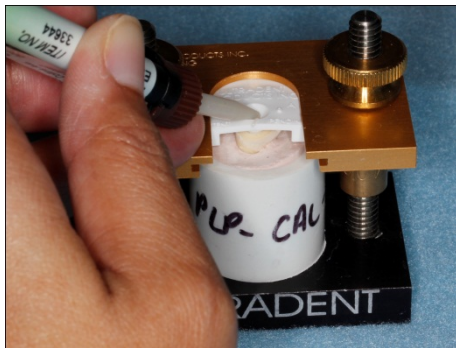
D- BluePhase Radiometer (Ivoclar)



E- Specimen mounted in the Ultradent Jig with Delrin Insert in place



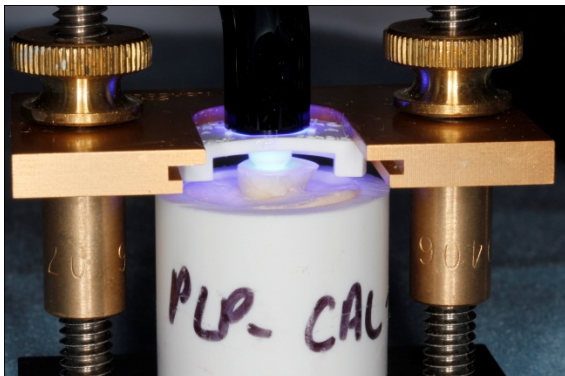
F- NX3 dual-cure resin cement syringed into Delrin Insert



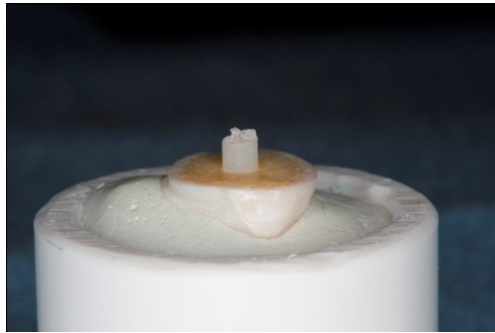
G- Calibra base and catalyst mixed and syringed into Delrin Insert



H- Cement being light cured



I- Cured specimen



J- Light proof box for initial storage of self-cure cement subgroups

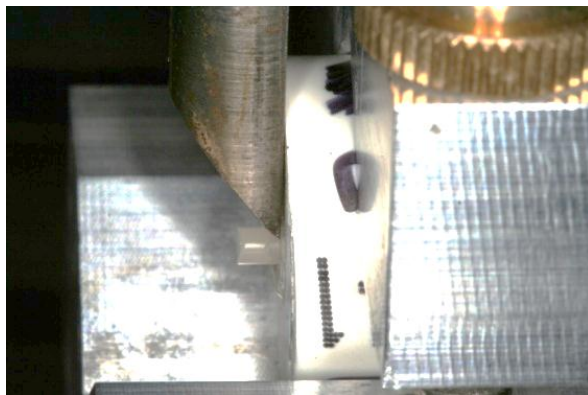


Figure 6 – Shear Bond Strength Testing

A- The Instron



B- Sample in Ultradent Jig with blade approximating specimen in preparation for testing



C. Statistical Management of Data

A mean and standard deviation was determined per group. Since this study involved a comparison between 16 subgroups, the appropriate test to implement was the ANOVA. Further the study involved three independent variables – adhesive (4 levels), cement (2 levels) and cure method (2 levels). Consequently, a three-way ANOVA was performed to identify differences at the three levels of variability. Alpha was set at 0.05.

Though significant differences were detected at all three levels with the three-way ANOVA, global conclusions could not be made from the results due to interactions between the individual parameters.

2-way ANOVAs were then performed, keeping one of the three variables constant each time. The alpha value was adjusted using the Bonferroni correction where the corrected alpha value was equal to the original value (0.05) divided by the number of tests within the particular parameter. A Tukey's Post Hoc test was used to determine differences between groups.

The first parameter evaluated was CEMENT. Since this had two levels, two, 2-way ANOVAs were performed with the corrected alpha value set at 0.025.

The second parameter was based on ADHESIVE. This had four levels so four, 2-way ANOVAs were performed with the alpha corrected to 0.0125.

IV. RESULTS

The statistical analysis was reviewed and approved by the clinical research administrator, Clinical Research Division, JBSA-Lackland, TX.

A. 2- WAY ANOVA Based on Adhesive (Table 3):

Four 2-way ANOVA tests were performed for the four levels tested within the ADHESIVES. The corrected $\alpha = 0.0125$.

Where significant differences were noted without interactions, a Tukey's Post Hoc test was performed to identify areas of difference.

Simplified Bonding Agent Group Results:

The simplified dental adhesives evaluated individually in this category included Prime and Bond NT (PB) and Prompt L Pop (PLP).

Within all the samples bonded with PB, irrespective of cure mode, no significant difference was noted between NX3 and CAL.

Within all the samples bonded with PLP, irrespective of cure mode, no significant difference was noted between NX3 and CAL.

Non-Simplified Dental Adhesive Results:

The non-simplified adhesives evaluated in this category included Clearfil SE (CSE) and Optibond FL (OFL).

Within all the samples bonded with OFL, irrespective of cure mode, NX3 bond strengths were significantly higher than that of Calibra.

Global Interpretation of Results:

When used with simplified bonding agents, NX3 bond strengths to dentin are not significantly higher than Calibra.

Table 4 – 2-Way ANOVA Based on Adhesive

| Cement | Shear Bond Strength (St dev) MPa | | | | | | | | | | | |
|--|----------------------------------|--------------|-------------|--------------|--------------|-------------|--------------|--------------|-------------|--------------|----------------|-------------|
| | Adhesive | | | | | | | | | | | |
| | Non-Simplified | | | | | | Simplified | | | | | |
| | CSE | | | OFL | | | PB | | | PLP | | |
| | DC | SC | 2-WAY ANOVA | DC | SC | 2-WAY ANOVA | DC | SC | 2-WAY ANOVA | DC | SC | 2-WAY ANOVA |
| NX3 | 13.2 (4.8) | 9.4 (3.2) | A | 6.5 (1.6) | 6.4 (1.7) | A | 2.3 (1.8) | 2.8 (2.0) | A | 2.5 (2.4) | 0.3 (0.3) | A |
| CAL | 9.5 (3.7) | 6.6 (4.2) | A | 4.0 (1.7) | 2.7 (2.4) | B | 3.8 (2.8) | 1.1 (1.2) | A | 1.1 (1.0) | 0.08 (0.04) | A |
| Groups with the same upper case letter per column within a bond group are not significantly different ($p > 0.0125$) | | | | | | | | | | | | |
| WHEN USED WITH SIMPLIFIED ADHESIVES, NX3 BOND STRENGTHS TO DENTIN ARE NOT SIGNIFICANTLY HIGHER THAN CALIBRA. | | | | | | | | | | | | |

B. 2-WAY ANOVA Based on Cement (Table 4):

Two, 2-way ANOVA tests were performed for the two levels tested within the CEMENTS. The corrected alpha = 0.025.

Within NX3 and Calibra, significant differences were noted at adhesive and cure mode levels. A Tukey's Post Hoc test was performed to identify areas of difference.

NX3 Results:

Within all the groups cemented with NX3, irrespective of adhesive used, the groups that were dual-cured showed higher bond strengths than those that were self-cured.

Within all the groups cemented with NX3, irrespective of cure-mode, samples bonded with non-simplified adhesives had significantly higher bond strengths than those bonded with the simplified adhesives. Among the adhesives, the CSE samples exhibited the highest bond strengths.

Calibra Results:

Within all the groups cemented with Calibra, irrespective of adhesive used, the groups that were dual-cured showed higher bond strengths than those that were self-cured.

Within all the groups cemented with Calibra, irrespective of cure-mode, samples cemented with the non-simplified adhesives showed higher bond strengths than the

simplified adhesives. It must be noted as an exception, the values shown by OFL were not significantly higher than those bonded with PB. Samples bonded with Clearfil SE exhibited the highest bond strengths.

Global Interpretation:

Dentin bond strengths are significantly higher when the cement (either NX3 or Calibra) is dual-cured.

Table 4 – 2-Way ANOVA Based on Cement

| Cure mode | Shear Bond Strength (Std dev) MPa Cement | | | | | | | | | |
|---|---|----------------|----------------|----------------|----------------|----------------|----------------|----------------|----------------|----------------|
| | NX3 | | | | | CAL | | | | |
| | CSE a | OFL b | PB c | PLP c | 2-WAY ANOVA | CSE a | OFL b | PB bc | PLP c | 2-WAY ANOVA |
| DC (Dual-cure) | 13.20 (4.78) | 6.49 (1.61) | 2.30 (1.76) | 2.45 (2.34) | A | 9.51 (3.74) | 3.97 (1.68) | 3.80 (2.85) | 1.07 (1.05) | A |
| SC (Self-Cure) | 9.43 (3.15) | 6.36 (1.69) | 2.81 (2.04) | 0.32 (0.34) | B | 6.58 (4.22) | 2.71 (2.37) | 1.13 (1.19) | 0.08 (0.04) | B |
| Groups with the same upper case letter per column within a cement group are not significantly different ($p>0.025$) Groups with the same lower case letter per row within a cement group are not significantly different ($p>0.025$) DENTIN BOND STRENGTHS ARE SIGNIFICANTLY HIGHER WHEN THE CEMENT (EITHER NX3 OR CALIBRA) IS DUAL CURED | | | | | | | | | | |

C. Fracture Mode Results (Figure 7):

Following de-bonding, all specimens were viewed under a 10x stereomicroscope to determine failure mode as either:

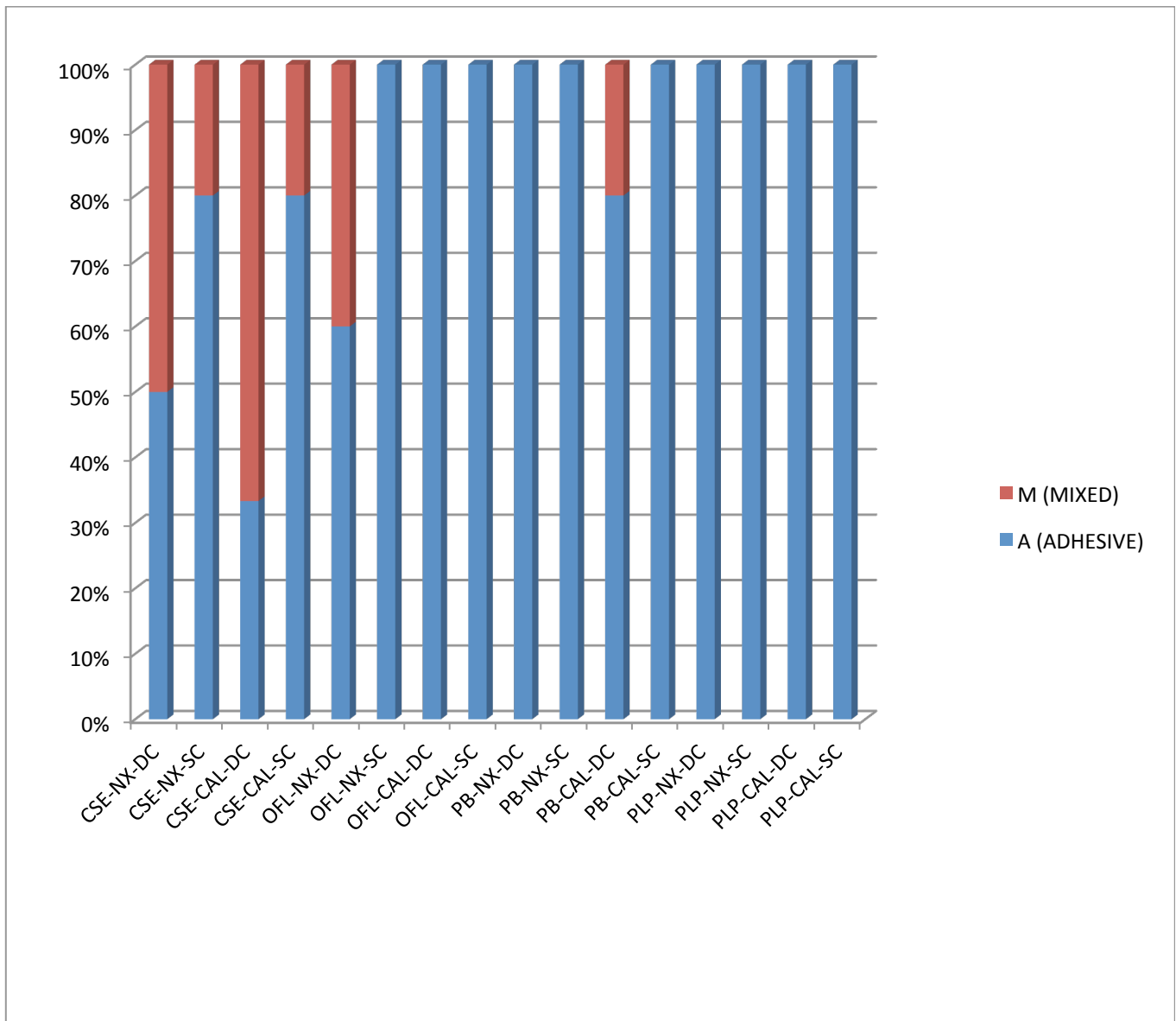
- 1) adhesive at cement/adhesive interface
- 2) cohesive within dentin or cement
- 3) mixed

The majority of the failures were adhesive.

No cohesive failures were noted.

Mixed failures were noted most in Clearfil SE Bond subgroups, suggesting greater adhesion. More mixed failures were associated with dual-curing.

Figure 7 - Fracture Mode Results



V. DISCUSSION (FIGURES 1,2)

A. OVERVIEW

“NX3 is compatible with self-etch and total-etch adhesives and obtains high bond strengths whether the cement is light or dual cured. A dual-cure activator for the adhesive is no longer needed and while light curing is always recommended even when there is limited light accessibility (i.e. PFM crowns); good adhesion is achieved in self-cure situations.”

(Kerr Press Release, 2007)

It was the aim of this study to test the validity of this claim. This study showed that when used specifically with simplified adhesives, either etch-and-rinse or self-etch, dentin bond strengths obtained are not significantly higher than Calibra. NX3 like every other dual-cure resin cement should therefore be used with a non-simplified adhesive for optimal results.

B. Overcoming the Incompatibilities between Simplified Dental Adhesives and Self-Cure Resin Cements (Figure 8)

The goal of patient-centered care is to provide excellent treatment in an efficient manner at minimum cost. With the development of stronger yet esthetic resin

materials and adhesives, we are getting closer to achieving this goal. It is no surprise that resin restorative materials and simplified adhesives are major players in the world of restorative dentistry. That being said, it is noteworthy that self- or dual-cure resin cements still maintain their place in any dentist's inventory. They are the norm in limited-light situations like composite-core build ups and cementation of posts and indirectly fabricated resin or ceramic crowns, inlays and onlays. However, incompatibilities between simplified dental adhesives and self-cure resins have been recognized as early as 1999 (Swift et al). Efforts at eliminating this problem can be targeted at three different levels.

1. Eliminate the acid-base reaction
2. Reduce the effects of the acid-base reaction
3. Eliminate the oxygen inhibited layer

The ideal solution would be one that permits continued use of the time saving simplified dental adhesive with any resin cement without compromising efficiency or bond strength.

The remainder of the discussion will explore these levels in detail and correlate them to the results of this study.

I. Eliminate the Acid-Base Reaction

This can be achieved in two ways: eliminating the acidic component or eliminating the base.

Eliminating the acidic component

One could avoid the acid component altogether by exclusive use of non-acidic non-simplified adhesives. This was re-confirmed by the results of our study, where we found that with either cement, in either cure mode, the non-simplified adhesives performed the best (Figures 1,2).

The alternative would be to mask the acid. This has been suggested by use of activators with the simplified adhesives. One commonly used product is the sodium salt of aryl sulphonic acid. This reacts with acidic resin monomers to produce phenyl or benzenesulphonyl free radicals that would then serve to initiate the polymerization of self-cured composites. However the dentin bond strengths obtained with these activators continues to be suboptimal (Tay, Suh et al, 2003). This is probably because the hydrophilicity of the acidic monomers is not overcome and osmotic blistering continues to be an issue (Tay et al, 2003). There is one study that examines the possibility of deprotonization of the acidic adhesive with an anion exchange compound with good results (Endo et al, 2007).

Eliminating the basic component

Tertiary amines are present in both light- and self-cure resin formulations. In light-cure resins, the light activates the camphorquinone initiator which is then transformed to its exciplex state by a tertiary amine accelerator. Self-cure systems employ a binary redox catalytic system composed of peroxide and a tertiary amine.

However, there is a difference. The tertiary amine in the light cure formulations are present in far less concentrations and are far less nucleophilic than those in self-cure formulations. This combined with the fact that the light-cure reaction takes place too fast to allow an acid-base reaction, account for the fact that the incompatibilities are restricted to self-cure groups.

The manufacturers of NX3 (Kerr) claim that the resin chemistry contains a unique redox system without need for the traditional tertiary amine. NX3 is referred to as a “universal cement” with universal applications. Unpublished abstracts presented by Kerr scientists claimed statistically higher bond strengths with various seventh generation bonding agents compared to Calibra and Variolink (Bui et al, IADR 2008). Another unpublished Kerr study stated that NX3 exhibited excellent bond compatibility with both etch-and-rinse and self-etch adhesives without need for an activator for the adhesive, resulting in simplified restorative procedures (Bui et al, AADR 2008). With the elimination of tertiary amines, the incompatibility issue would be solved and dentists would be free to use NX3 with any adhesive of their choice.

How accurate is this claim? A literature search revealed no published studies thus far. A recent unpublished poster presentation by Dentsply at the 2010 IADR meeting compared bond strengths achieved with Smart Cem 2 (self-cure cement from Dentsply) and NX3 with various simplified dental adhesives with NX3 showing the lowest values (Liu et al, IADR 2010). Another unpublished presentation by the Tokuyama Corporation, compared their experimental simplified dentin adhesive

agent with various self-cure resin cements to include NX3, Calibra and Clearfil Esthetic Cement, with the result that bond strengths with NX3 were not statistically different than any other cement in that category (Hirata et al, IADR 2012). Finally, an abstract submitted for presentation at the 2013 IADR by Bisco Inc. compared their simplified adhesive and self-cure resin combination All-Bond Universal and Duolink with Kerr's Optibond All-in-One and NX3, with both showing no statistically significant difference (Chen et al, IADR 2013).

The results of our study show that although overall dentin bond strengths with NX3 appear superior to Calibra when considered globally irrespective of adhesive or cure mode, when looked at specifically within the realm of simplified adhesives, the bond strengths with NX3 are not statistically higher than Calibra. The proprietary redox system is a trade secret and the exact chemistry is not available in the NX3 MSDS. Whatever the composition, it is apparent that it does not completely overcome the incompatibilities. NX3 appears no different from other dual-cure resin cements on the market. The numeric bond strength values are higher than Calibra most likely because the absence of tertiary amines allowed for more complete polymerization of the resin. However, the presence of residual uncured hydrophilic acidic monomers at the oxygen inhibited interface could continue to contribute to interfacial stresses and less than optimal bond strengths.

Null Hypothesis #1: There is no difference in shear bond strength to dentin of NX3 or Calibra when used with Simplified Dentin Adhesive Agents

Despite manufacturer's claims, we failed to reject the null hypothesis.

II. Reduce the Effects of the Acid-Base Reaction: Light Cure Adequately

When using a dual-cure composite, if clinical conditions allow, adequate light curing of the cement is sufficient to overcome the acid-base interactions (Tay et al, 2003). The bond strength to dentin is directly related to the amount of light energy to which it is exposed (Takahashi et al, 2010). Photo polymerization results in rapid setting of the resin matrix, allowing no time for adverse acid-base reactions to occur. The light energy should also be able to successfully and rapidly cure the acidic monomers in the oxygen inhibited layer with remnants of the photo-initiator (Suh, 2004). Results of the 2-way ANOVA confirmed that with either resin cement NX3 or Calibra, the dentin bond strengths were significantly higher when the cement is dual-cured.

Null Hypothesis #2: There is no difference in the shear bond strength to dentin of the resin cements based on the curing mode. This null hypothesis was rejected.

III. Eliminate the Oxygen Inhibited Layer

Light and chemically-cured dental composite resins leave a soft, sticky superficial layer upon polymerization. When oxygen diffuses through the superficial layer of resin, it forms peroxide radicals with the monomer. The peroxide radicals are poorly reactive and do not participate in the polymerization reaction. This layer has

the same composition as the uncured resin except it has less of the photo-initiator (Eliades et al, 1989).

This layer in simplified adhesives serves both as a source of acidic resin monomers that deactivate the tertiary amines and as a hypertonic medium that triggers osmotic fluid transport through the adhesive layer (Suh et al, 2003).

How can we eliminate this layer? The answer is two-fold. The oxygen inhibited layer can be prevented or it can be removed after formation.

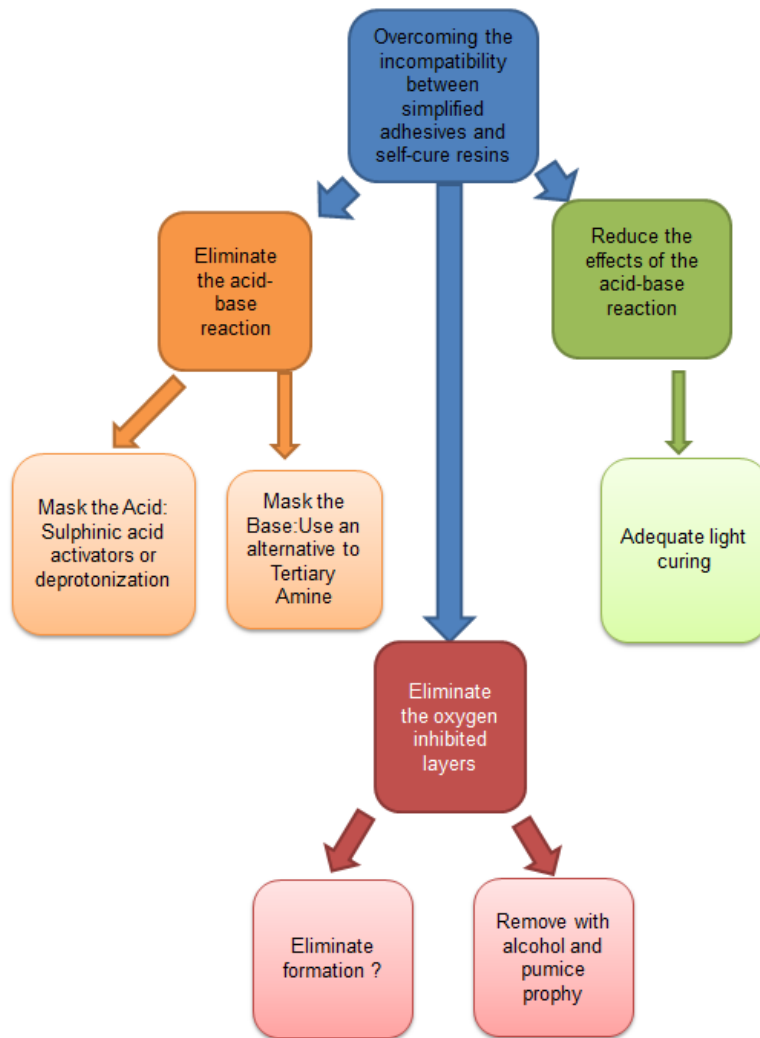
Preventing formation of the oxygen inhibited layer can be achieved by polymerization in an inert nitrogen environment (not clinically feasible) or coating adhesive with glycerol prior to activation.

Removal of the oxygen inhibited layer has been suggested by wiping/rubbing with isopropyl alcohol or prophylaxis with prophyl cup with a mixture of flour of pumice and rubbing alcohol (<http://leeannbrady.com/restorative-dentistry/removing-the-air-inhibited-layer>). Since dentin adhesive agents are applied in thin layers unlike restorative composites, the use of the latter technique could remove too much of the bonding agent, creating direct contact of resin with the hybridized dentin (Suh et al, 2003).

It is noteworthy that contrary to common perception, presence of an oxygen inhibited layer is not required for higher bond strengths to additional increments of composite (Ghivari et al 2009, Rueggerberg et al 1990, Eliades et al 1989).

In spite of the sound theory behind elimination of the oxygen inhibited layer, a study evaluating its efficacy in improving bond incompatibilities of self-etch adhesives on self-cure resins, showed persistence of low bond strengths, irrespective of its presence (Endo et al, 2007). This study was very limited in sample size (1 sample per group) and perhaps warrants additional research.

Table 8 - Treatment Strategies Flow Chart



VI. CONCLUSION

In conclusion, within the limitations of this study the following recommendations can be made:

- When used specifically with simplified dental adhesives in either cure mode, NX3 does not produce significantly higher bond strengths than Calibra.
- In general, lower bond strengths continue to be observed when simplified adhesives are used with the resin cements in self-cure mode. Continue to restrict usage with limited light-cure situations.
- Dentin bond strengths can be maximized when the resin cement is dual cured.
- Clearfil SE Bond shows superior bond strengths with the resin cements in any cure mode

VII. DISCLOSURES:

The views expressed in this manuscript express those of the authors and do not reflect the official policy of the Department of Defense or other departments of the United States Government. The authors do not have any financial interest in the companies whose materials are discussed in the document.

VIII. Appendix A- Raw Data by Group

Legend

Treatment groups:

NX/NX3- Nexus 3

CAL- Calibra

OFL- Optibond FL

CSE- Clearfil SE

PB- Prime and Bond NT

PLP- Prompt L- Pop

SC- Selfcure

LC- Light cure

Subgroup 1- PB-NX-SC

| | | |
|----------------|--------|------|
| Prime and Bond | | |
| NT | | |
| NX SC | | |
| | N | MPa |
| 1 | 25.840 | 5.76 |
| 2 | 1.390 | 0.31 |
| 3 | 17.860 | 3.98 |
| 4 | 19.210 | 4.28 |
| 5 | 9.420 | 2.09 |
| 6 | 5.200 | 1.16 |
| 7 | 8.690 | 1.94 |
| 8 | 5.530 | 1.23 |
| 9 | 27.000 | 6.02 |
| 10 | 5.860 | 1.31 |
| avg | | 2.81 |
| stdv | | 2.04 |

Subgroup 2-PB-NX-LC

NX LC

| | N | MPa |
|------|--------|------|
| 1 | 10.930 | 2.44 |
| 2 | 21.210 | 4.73 |
| 3 | 0.870 | 0.19 |
| 4 | 12.040 | 2.68 |
| 5 | 3.190 | 0.71 |
| 6 | 5.150 | 1.15 |
| 7 | 13.330 | 2.97 |
| 8 | 22.610 | 5.04 |
| 9 | 0.260 | 0.06 |
| 10 | 13.830 | 3.08 |
| avg | | 2.31 |
| stdv | | 1.76 |

Subgroup 3- PB-CAL-SC

Cal SC

| | N | MPa |
|------|--------|------|
| 1 | 9.010 | 2.00 |
| 2 | 0.00 | 0.00 |
| 3 | 3.360 | 0.75 |
| 4 | 0.170 | 0.04 |
| 5 | 0.760 | 0.17 |
| 6 | 4.470 | 1.00 |
| 7 | 0.00 | 0.00 |
| 8 | 14.580 | 3.25 |
| 9 | 12.470 | 2.78 |
| 10 | 3.350 | 1.34 |
| avg | | 1.13 |
| stdv | | 1.19 |

Subgroup 4- PB-CAL-LC

Cal LC

| | N | MPa |
|------|--------|------|
| 1 | 4.500 | 1.00 |
| 2 | 5.280 | 1.17 |
| 3 | 33.560 | 7.48 |
| 4 | 0.00 | 0.00 |
| 5 | 27.100 | 6.04 |
| 6 | 3.830 | 0.85 |
| 7 | 34.310 | 7.65 |
| 8 | 21.150 | 4.71 |
| 9 | 20.650 | 4.60 |
| 10 | 20.190 | 4.50 |
| avg | | 3.80 |
| stdv | | 2.85 |

Subgroup 5- PLP-NX-SC

NX SC

| | N | MPa |
|------|-------|------|
| 1 | 0.690 | 0.15 |
| 2 | 5.685 | 1.27 |
| 3 | 1.320 | 0.29 |
| 4 | 0.820 | 0.18 |
| 5 | 0.710 | 0.16 |
| 6 | 1.410 | 0.31 |
| 7 | 0.890 | 0.20 |
| 8 | 0.690 | 0.15 |
| 9 | 1.330 | 0.30 |
| 10 | 0.610 | 0.14 |
| avg | | 0.32 |
| stdv | | 0.34 |

Subgroup 6- PLP-NX-LC

NX LC

| | N | MPa |
|------|--------|------|
| 1 | 4.460 | 1.00 |
| 2 | 0.410 | 0.09 |
| 3 | 5.060 | 1.13 |
| 4 | 8.180 | 1.82 |
| 5 | 39.470 | 8.80 |
| 6 | 12.300 | 2.74 |
| 7 | 12.160 | 2.71 |
| 8 | 6.060 | 1.35 |
| 9 | 12.270 | 2.74 |
| 10 | 9.450 | 2.11 |
| avg | | 2.45 |
| stdv | | 2.40 |

Subgroup 7- PLP-CAL-SC

Cal SC

| | N | MPa |
|------|-------|------|
| 1 | 0.220 | 0.05 |
| 2 | 0.330 | 0.07 |
| 3 | 0.560 | 0.12 |
| 4 | 0.240 | 0.05 |
| 5 | 0.290 | 0.07 |
| 6 | 0.390 | 0.09 |
| 7 | 0.470 | 0.11 |
| 8 | 0.260 | 0.06 |
| 9 | 0.200 | 0.05 |
| 10 | 0.770 | 0.17 |
| avg | | 0.08 |
| stdv | | 0.04 |

Subgroup 8- PLP-CAL-LC

Cal LC

| | N | MPa |
|------|--------|------|
| 1 | 13.520 | 3.01 |
| 2 | 2.000 | 0.45 |
| 3 | 0.320 | 0.07 |
| 4 | 1.230 | 0.27 |
| 5 | 0.560 | 0.12 |
| 6 | 1.930 | 0.43 |
| 7 | 3.290 | 0.73 |
| 8 | 7.230 | 1.60 |
| 9 | 11.500 | 2.56 |
| 10 | 6.400 | 1.43 |
| avg | | 1.07 |
| stdv | | 1.05 |

Subgroup 9- OFL-NX-SC

Optibond FL

NX SC

| | N | MPa |
|------|--------|------|
| | 35.240 | 7.99 |
| | 39.640 | 8.99 |
| | 26.830 | 6.08 |
| | 28.160 | 6.39 |
| | 28.840 | 6.54 |
| | 19.650 | 4.46 |
| | 20.690 | 4.69 |
| | 36.620 | 8.30 |
| | 27.100 | 6.15 |
| | 17.530 | 3.98 |
| avg | | 6.36 |
| stdv | | 1.69 |

Subgroup 10- OFL-NX-LC

NX LC

| | N | MPa |
|------|--------|------|
| 1 | 27.600 | 6.15 |
| 2 | 40.400 | 9.01 |
| 3 | 20.200 | 4.50 |
| 4 | 36.890 | 8.22 |
| 5 | 22.340 | 4.98 |
| 6 | 27.700 | 6.17 |
| 7 | 22.530 | 5.02 |
| 8 | 25.040 | 5.58 |
| 9 | 45.920 | 6.65 |
| 10 | 38.650 | 8.61 |
| avg | | 6.49 |
| stdv | | 1.61 |

Subgroup 11- OFL-CAL-SC

Cal SC

| | N | MPa |
|------|--------|------|
| 1 | 38.850 | 8.81 |
| 2 | 7.800 | 1.77 |
| 3 | 14.210 | 3.22 |
| 4 | 1.260 | 0.29 |
| 5 | 8.450 | 1.92 |
| 6 | 11.840 | 2.68 |
| 7 | 14.830 | 3.36 |
| 8 | 4.680 | 1.06 |
| 9 | 4.780 | 1.08 |
| 10 | 12.960 | 2.94 |
| avg | | 2.71 |
| stdv | | 2.37 |

Subgroup 12- OFL-CAL-LC

Cal LC

| | N | MPa |
|------|--------|------|
| 1 | 20.170 | 4.49 |
| 2 | 24.270 | 5.41 |
| 3 | 26.430 | 5.89 |
| 4 | 28.350 | 6.32 |
| 5 | 9.160 | 2.04 |
| 6 | 21.980 | 4.90 |
| 7 | 17.500 | 3.90 |
| 8 | 8.450 | 1.88 |
| 9 | 10.030 | 2.24 |
| 10 | 11.900 | 2.65 |
| avg | | 3.97 |
| stdv | | 1.68 |

Subgroup 13- CSE-NX-SC

Clearfil SE

NX SC

| | N | MPa |
|------|--------|-------|
| | 13.390 | 2.98 |
| | 34.240 | 7.63 |
| | 64.610 | 14.40 |
| | 32.280 | 7.19 |
| | 53.020 | 11.81 |
| | 51.500 | 11.68 |
| | 47.540 | 10.60 |
| | 48.470 | 10.59 |
| | 38.160 | 8.51 |
| | 39.920 | 8.90 |
| avg | | 9.43 |
| stdv | | 3.15 |

Subgroup 14- CSE-NX-LC

NX LC

| | N | MPa |
|------|--------|-------|
| 1 | 81.160 | 18.09 |
| 2 | 40.600 | 9.05 |
| 3 | 42.000 | 9.35 |
| 4 | 87.800 | 19.57 |
| 5 | 36.730 | 8.19 |
| 6 | 65.370 | 14.57 |
| 7 | 57.940 | 12.91 |
| 8 | 73.410 | 16.36 |
| 9 | 80.250 | 17.88 |
| 10 | 26.930 | 6.11 |
| avg | | 13.21 |
| stdv | | 4.78 |

Subgroup 15- CSE-CAL-SC

Cal SC

| | N | MPa |
|------|--------|-------|
| 1 | 5.080 | 1.13 |
| 2 | 29.050 | 6.48 |
| 3 | 72.460 | 16.15 |
| 4 | 11.570 | 2.58 |
| 5 | 32.540 | 7.25 |
| 6 | 20.100 | 4.48 |
| 7 | 25.970 | 5.89 |
| 8 | 41.450 | 9.40 |
| 9 | 17.640 | 4.00 |
| 10 | 37.810 | 8.43 |
| avg | | 6.58 |
| stdv | | 4.23 |

Subgroup 16-CSE-CAL-LC

Cal LC

| | N | MPa |
|------|--------|-------|
| 1 | 63.160 | 14.08 |
| 2 | 15.760 | 3.51 |
| 3 | 55.370 | 12.34 |
| 4 | 47.150 | 10.51 |
| 5 | 60.610 | 13.51 |
| 6 | 46.830 | 10.44 |
| 7 | 14.960 | 3.34 |
| 8 | 43.840 | 9.77 |
| 9 | 45.590 | 10.16 |
| 10 | 33.280 | 7.42 |
| avg | | 9.51 |
| stdv | | 3.74 |

Appendix B - Statistical Analysis

2-Way ANOVA BASED ON CEMENT:

2 Way ANOVA-CEMENT- CAL

| Descriptive Statistics Dependent Variable: MPA | | | | |
|---|-------|-----------|----------------|----|
| BOND | MODE | Mean | Std. Deviation | N |
| CSE | LC | 9.5080 | 3.7370 | 10 |
| | SC | 6.5790 | 4.2251 | 10 |
| | Total | 8.0435 | 4.1628 | 20 |
| Opti | LC | 3.9720 | 1.6750 | 10 |
| | SC | 2.7130 | 2.3732 | 10 |
| | Total | 3.3425 | 2.1010 | 20 |
| PB | LC | 3.8000 | 2.8527 | 10 |
| | SC | 1.1330 | 1.1946 | 10 |
| | Total | 2.4665 | 2.5303 | 20 |
| Pop | LC | 1.0670 | 1.0460 | 10 |
| | SC | 8.400E-02 | 3.921E-02 | 10 |
| | Total | .5755 | .8793 | 20 |
| Total | LC | 4.5868 | 3.9551 | 40 |
| | SC | 2.6273 | 3.4621 | 40 |
| | Total | 3.6070 | 3.8225 | 80 |

| Tests of Between-Subjects Effects Dependent Variable: MPA | | | | | |
|--|-------------------------|----|-------------|---------|------|
| Source | Type III Sum of Squares | df | Mean Square | F | Sig. |
| Corrected Model | 696.081(a) | 7 | 99.440 | 15.625 | .000 |
| Intercept | 1040.836 | 1 | 1040.836 | 163.542 | .000 |
| BOND | 604.864 | 3 | 201.621 | 31.680 | .000 |
| MODE | 76.793 | 1 | 76.793 | 12.066 | .001 |
| BOND * MODE | 14.424 | 3 | 4.808 | .755 | .523 |
| Error | 458.233 | 72 | 6.364 | | |
| Total | 2195.150 | 80 | | | |
| Corrected Total | 1154.314 | 79 | | | |
| a R Squared = .603 (Adjusted R Squared = .564) | | | | | |

Homogeneous Subsets

| MPA Tukey HSD | | | | |
|---|----|--------|--------|--------|
| | N | Subset | | |
| BOND | | 1 | 2 | 3 |
| Pop | 20 | .5755 | | |
| PB | 20 | 2.4665 | 2.4665 | |
| Opti | 20 | | 3.3425 | |
| CSE | 20 | | | 8.0435 |
| Sig. | | .092 | .692 | 1.000 |
| Means for groups in homogeneous subsets are displayed. Based on Type III Sum of Squares The error term is Mean Square(Error) = 6.364. | | | | |
| a Uses Harmonic Mean Sample Size = 20.000. | | | | |
| b Alpha = .05. | | | | |

2 Way ANOVA-CEMENT- NX3

Tests of Between-Subjects Effects

Dependent Variable: MPA

| Source | Type III Sum of Squares | df | Mean Square | F | Sig. |
|-----------------|-------------------------|----|-------------|---------|------|
| Corrected Model | 1301.594(a) | 7 | 185.942 | 28.979 | .000 |
| Intercept | 2350.112 | 1 | 2350.112 | 366.267 | .000 |
| BOND | 1206.068 | 3 | 402.023 | 62.656 | .000 |
| MODE | 38.392 | 1 | 38.392 | 5.983 | .017 |
| BOND * MODE | 57.134 | 3 | 19.045 | 2.968 | .038 |
| Error | 461.980 | 72 | 6.416 | | |
| Total | 4113.686 | 80 | | | |
| Corrected Total | 1763.574 | 79 | | | |

a R Squared = .738 (Adjusted R Squared = .713)

Homogeneous Subsets

MPA
Tukey HSD

| | N | Subset | | |
|------|----|--------|--------|---------|
| BOND | | 1 | 2 | 3 |
| Pop | 20 | 1.3820 | | |
| PB | 20 | 2.5565 | | |
| Opti | 20 | | 6.4230 | |
| CSE | 20 | | | 11.3185 |
| Sig. | | .463 | 1.000 | 1.000 |

Means for groups in homogeneous subsets are displayed.
Based on Type III Sum of Squares
The error term is Mean Square(Error) = 6.416.

a Uses Harmonic Mean Sample Size = 20.000.

b Alpha = .05.

2-way cement vs mode

| Descriptive Statistics Dependent Variable: MPA | | | | |
|---|-------|--------|----------------|-----|
| CEMENT | MODE | Mean | Std. Deviation | N |
| Cal | LC | 4.5868 | 3.9551 | 40 |
| | SC | 2.6273 | 3.4621 | 40 |
| | Total | 3.6070 | 3.8225 | 80 |
| NX | LC | 6.1127 | 5.2934 | 40 |
| | SC | 4.7273 | 4.0269 | 40 |
| | Total | 5.4200 | 4.7248 | 80 |
| Total | LC | 5.3498 | 4.7058 | 80 |
| | SC | 3.6773 | 3.8780 | 80 |
| | Total | 4.5135 | 4.3793 | 160 |

| Tests of Between-Subjects Effects Dependent Variable: MPA | | | | | |
|--|-------------------------|-----|-------------|---------|------|
| Source | Type III Sum of Squares | df | Mean Square | F | Sig. |
| Corrected Model | 246.664(a) | 3 | 82.221 | 4.576 | .004 |
| Intercept | 3259.469 | 1 | 3259.469 | 181.424 | .000 |
| CEMENT | 131.479 | 1 | 131.479 | 7.318 | .008 |
| MODE | 111.890 | 1 | 111.890 | 6.228 | .014 |
| CEMENT * MODE | 3.295 | 1 | 3.295 | .183 | .669 |
| Error | 2802.703 | 156 | 17.966 | | |
| Total | 6308.836 | 160 | | | |
| Corrected Total | 3049.367 | 159 | | | |
| a R Squared = .081 (Adjusted R Squared = .063) | | | | | |

2-WAY ANOVA BASED ON ADHESIVE:

2-way PB

| Descriptive Statistics | | | | |
|-------------------------|-------|--------|----------------|----|
| Dependent Variable: MPA | | | | |
| CEMENT | MODE | Mean | Std. Deviation | N |
| Cal | LC | 3.8000 | 2.8527 | 10 |
| | SC | 1.1330 | 1.1946 | 10 |
| | Total | 2.4665 | 2.5303 | 20 |
| NX | LC | 2.3050 | 1.7624 | 10 |
| | SC | 2.8080 | 2.0421 | 10 |
| | Total | 2.5565 | 1.8743 | 20 |
| Total | LC | 3.0525 | 2.4319 | 20 |
| | SC | 1.9705 | 1.8411 | 20 |
| | Total | 2.5115 | 2.1984 | 40 |

| Tests of Between-Subjects Effects | | | | | |
|-----------------------------------|-------------------------|----|-------------|--------|------|
| Dependent Variable: MPA | | | | | |
| Source | Type III Sum of Squares | df | Mean Square | F | Sig. |
| Corrected Model | 36.910(a) | 3 | 12.303 | 2.922 | .047 |
| Intercept | 252.305 | 1 | 252.305 | 59.927 | .000 |
| CEMENT | 8.100E-02 | 1 | 8.100E-02 | .019 | .890 |
| MODE | 11.707 | 1 | 11.707 | 2.781 | .104 |
| CEMENT * MODE | 25.122 | 1 | 25.122 | 5.967 | .020 |
| Error | 151.567 | 36 | 4.210 | | |
| Total | 440.783 | 40 | | | |
| Corrected Total | 188.478 | 39 | | | |

a R Squared = .196 (Adjusted R Squared = .129)

2-way PLP

| Descriptive Statistics Dependent Variable: MPA | | | | |
|---|-------|-----------|----------------|----|
| CEMENT | MODE | Mean | Std. Deviation | N |
| Cal | LC | 1.0670 | 1.0460 | 10 |
| | SC | 8.400E-02 | 3.921E-02 | 10 |
| | Total | .5755 | .8793 | 20 |
| NX | LC | 2.4490 | 2.3966 | 10 |
| | SC | .3150 | .3421 | 10 |
| | Total | 1.3820 | 1.9937 | 20 |
| Total | LC | 1.7580 | 1.9343 | 20 |
| | SC | .1995 | .2650 | 20 |
| | Total | .9787 | 1.5748 | 40 |

| Tests of Between-Subjects Effects Dependent Variable: MPA | | | | | |
|--|-------------------------|----|-------------|--------|------|
| Source | Type III Sum of Squares | df | Mean Square | F | Sig. |
| Corrected Model | 34.106(a) | 3 | 11.369 | 6.537 | .001 |
| Intercept | 38.318 | 1 | 38.318 | 22.033 | .000 |
| CEMENT | 6.504 | 1 | 6.504 | 3.740 | .061 |
| MODE | 24.289 | 1 | 24.289 | 13.966 | .001 |
| CEMENT * MODE | 3.312 | 1 | 3.312 | 1.904 | .176 |
| Error | 62.609 | 36 | 1.739 | | |
| Total | 135.033 | 40 | | | |
| Corrected Total | 96.714 | 39 | | | |
| a R Squared = .353 (Adjusted R Squared = .299) | | | | | |

2-way OFL

Descriptive Statistics
Dependent Variable: MPA

| CEMENT | MODE | Mean | Std. Deviation | N |
|--------|-------|--------|----------------|----|
| Cal | LC | 3.9720 | 1.6750 | 10 |
| | SC | 2.7130 | 2.3732 | 10 |
| | Total | 3.3425 | 2.1010 | 20 |
| NX | LC | 6.4890 | 1.6094 | 10 |
| | SC | 6.3570 | 1.6851 | 10 |
| | Total | 6.4230 | 1.6052 | 20 |
| Total | LC | 5.2305 | 2.0550 | 20 |
| | SC | 4.5350 | 2.7400 | 20 |
| | Total | 4.8828 | 2.4164 | 40 |

Tests of Between-Subjects Effects

Dependent Variable: MPA

| Source | Type III Sum of Squares | df | Mean Square | F | Sig. |
|-----------------|-------------------------|----|-------------|---------|------|
| Corrected Model | 102.907(a) | 3 | 34.302 | 9.894 | .000 |
| Intercept | 953.650 | 1 | 953.650 | 275.074 | .000 |
| CEMENT | 94.895 | 1 | 94.895 | 27.372 | .000 |
| MODE | 4.837 | 1 | 4.837 | 1.395 | .245 |
| CEMENT * MODE | 3.175 | 1 | 3.175 | .916 | .345 |
| Error | 124.808 | 36 | 3.467 | | |
| Total | 1181.365 | 40 | | | |
| Corrected Total | 227.715 | 39 | | | |

a R Squared = .452 (Adjusted R Squared = .406)

2-way CSE

| Descriptive Statistics | | | | |
|-------------------------|-------|---------|----------------|----|
| Dependent Variable: MPA | | | | |
| CEMENT | MODE | Mean | Std. Deviation | N |
| Cal | LC | 9.5080 | 3.7370 | 10 |
| | SC | 6.5790 | 4.2251 | 10 |
| | Total | 8.0435 | 4.1628 | 20 |
| NX | LC | 13.2080 | 4.7813 | 10 |
| | SC | 9.4290 | 3.1470 | 10 |
| | Total | 11.3185 | 4.3907 | 20 |
| Total | LC | 11.3580 | 4.5877 | 20 |
| | SC | 8.0040 | 3.9095 | 20 |
| | Total | 9.6810 | 4.5370 | 40 |

| Tests of Between-Subjects Effects | | | | | |
|--|-------------------------|----|-------------|---------|------|
| Dependent Variable: MPA | | | | | |
| Source | Type III Sum of Squares | df | Mean Square | F | Sig. |
| Corrected Model | 221.556(a) | 3 | 73.852 | 4.574 | .008 |
| Intercept | 3748.870 | 1 | 3748.870 | 232.196 | .000 |
| CEMENT | 107.256 | 1 | 107.256 | 6.643 | .014 |
| MODE | 112.493 | 1 | 112.493 | 6.968 | .012 |
| CEMENT * MODE | 1.806 | 1 | 1.806 | .112 | .740 |
| Error | 581.230 | 36 | 16.145 | | |
| Total | 4551.656 | 40 | | | |
| Corrected Total | 802.785 | 39 | | | |
| a R Squared = .276 (Adjusted R Squared = .216) | | | | | |

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